



(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:  
**21.04.2010 Bulletin 2010/16**

(51) Int Cl.:  
**A23L 3/3445** (2006.01) **A23B 9/22** (2006.01)  
**A01N 59/02** (2006.01) **A01N 59/04** (2006.01)  
**A01P 7/04** (2006.01)

(21) Application number: **09170684.6**

(22) Date of filing: **18.09.2009**

(84) Designated Contracting States:  
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO SE SI SK SM TR**  
Designated Extension States:  
**AL BA RS**

- **Gabarra, Rosa**  
**08348 Cabrils (ES)**
- **Castañé, Cristina**  
**08348 Cabrils (ES)**
- **Alomar, Oscar**  
**08348 Cabrils (ES)**
- **Guri, Sonia**  
**08193 Bellaterra (ES)**
- **Sánchez, Javier**  
**08009 Barcelona (ES)**

(30) Priority: **19.09.2008 EP 08382036**

(71) Applicant: **Sociedad española de carburos metálicos, S.A.**  
**08009 Barcelona (ES)**

(72) Inventors:  
• **Riudavets, Jordi**  
**08348 Cabrils (ES)**

(74) Representative: **Ponti Sales, Adelaida**  
**Oficina Ponti**  
**C. Consell de Cent 322**  
**08007 Barcelona (ES)**

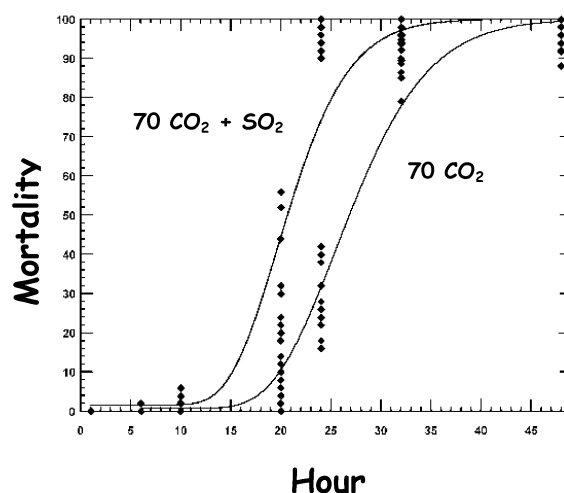
(54) **Process for eradication of insects and/or mites at any stage of their development in an agricultural product**

(57) The process of the invention comprises the steps of a) placing the agricultural product in a container; b) filling the container with a mixture of gases comprising CO<sub>2</sub>, O<sub>2</sub> and SO<sub>2</sub>, by means of flushing the mixture of gases or by means of creating a vacuum inside the container; and c) exposing the agricultural product to said mixture for a time ranging between 4 hours and 20 days, so that it permits the eradication of insects at any stage of their development.

This mixture comprises a concentration of CO<sub>2</sub> higher than 40% and a concentration of O<sub>2</sub> ranging from 0.5% to 10.5%; and a concentration of SO<sub>2</sub> ranging from 50ppm to 30, which mixture is balanced with an inert gas.

This process allows to significantly reducing treatment time to eradicate different insects in all of their development stages.

**FIG. 1**



**Description****FIELD OF THE INVENTION**

[0001] This invention relates to a process for eradication of a wide range of insects and/or mites in an agricultural product, particularly it relates to a process for the eradication of insects and/or mites in all types of agricultural products susceptible to be infected by an insect and/or mite.

[0002] This process allows to significantly reduce treatment time to eradicate different insects in all of their development stages being said agricultural products a cultivated product or a manufactured product such as a stored non-perishable food product, and the like.

**BACKGROUND OF THE INVENTION**

[0003] Modified atmospheres (MA) with a high carbon dioxide (CO<sub>2</sub>) content are safe and environmentally friendly pest control process for raw and manufactured products.

[0004] Methyl bromide is a fumigant that has been used commercially for more than 50 years to control pests. The Montreal Protocol decided to phase it out on January 2005, particularly for foods products. For that reason, researchers have to find other solution to control pest in food products. Carbon dioxide is a good alternative to methyl bromide, phosphine and other treatments because no residues in final product, is a food additive, and no safety interval after the treatment is required.

[0005] The main advantages of using high CO<sub>2</sub> content in a MA are that they are effective for the control of a wide range of pest species, and they can be used for the treatment of different products without an accumulation of toxic residues after the treatment. However, for example, control of insects in processed agricultural product by means of MA is sometimes too long for the marketing requirements of the industry. Data on the effects of different CO<sub>2</sub> types of treatments and dosages are available for many species and stages of stored product pests under many particular sets of conditions. The recommended exposure time to achieve complete pest control at the most appropriate gas concentration may take from several days to weeks, according to CO<sub>2</sub> concentration, developmental stage and species. Thus, with high carbon dioxide treatment, at atmospheric pressure, the time needed to kill insects is approximately 21 days (it depends on the kind of insect and insect stage).

[0006] On the other hand, sulphur dioxide (SO<sub>2</sub>) is a gas accepted as a food additive (E-220). It has a large history as a food preservative due to its antimicrobial properties in a range of food products and beverages. Official dosages approved for the treatment of food products may range from 50ppm in cereal by-products, to 500ppm in nuts and 2000ppm in dried fruits.

[0007] Also, DE19506631 describes a treatment for fumigating objects and devices which have been attacked by pests introducing a gas current containing CO<sub>2</sub> and a toxic gas into a place where products to be treated are confined. CO<sub>2</sub> helps the toxic gas penetrate inside the insect eggs and pupae and its concentration is between 5% and 30%. There is not described nor suggested a particular effect of oxygen gas in the toxic mixture.

[0008] Furthermore, US6047496 discloses a process for structural fumigation using CO<sub>2</sub> as the carrier of the gaseous toxic agent. The use of CO<sub>2</sub> as carrier allows a reduction of the concentration of toxic agent and the treatment times. The useful toxic agents methyl bromide, sulfuryl fluoride and sulfur dioxide are cited. Again, the concentration of CO<sub>2</sub> is not higher than 20%, neither a particular effect of oxygen gas in the toxic mixture is described.

[0009] Thus, the objective of this invention is to provide a process for the eradication of pests which in the present invention refers to insects and/or mites in an agricultural product wherein the exposure time be substantially reduced.

**BRIEF DESCRIPTION OF THE INVENTION**

[0010] This invention relates to a process for eradication of insects and/or mites at any stage of their development in an agricultural product, **characterized in that** it comprises the steps of:

- a) placing the agricultural product inside of a container;
- b) filling the container with a mixture of gases consisting of CO<sub>2</sub>, O<sub>2</sub>, SO<sub>2</sub>, and an inert gas, wherein the concentration of the CO<sub>2</sub> is higher than 40%, the O<sub>2</sub> is between 0.5 to 10.5%, the SO<sub>2</sub> is between 50 ppm to 3%, and balanced with an inert gas, by means of flushing the mixture of gases or by means of creating a vacuum inside the container then filling the container with said mixture;
- c) exposing the agricultural product to the mixture of gases for a time that ranges between 4 hours and 20 days, so that it permits the eradication of insects and/or mites at any stage of their development inside of the container.

[0011] It should be understood by a skilled person in the art that each component of the mixture may include impurities

at trace level which are not involved in the eradication process.

**[0012]** Preferably, the steps b) and c) are carried out at atmospheric pressure and at room temperature

**[0013]** In this invention, "flushing" is taken to mean displacing the air atmosphere inside the container by introducing a stream of the mixture of gases consisting of CO<sub>2</sub>, O<sub>2</sub> and SO<sub>2</sub>, previously prepared.

**[0014]** In this invention, the term "agricultural product" refers to a product of plant or animal origin, whether it be a cultivated product or a manufactured product, such as a stored non-perishable food product, for example, cereal products or dried fruits, such as a wood product, or such as other non-perishable products like the cotton, or the tobacco, etc.

**[0015]** The object of the present invention is to provide a process for eradication of insects and/or mites in an agricultural product and thus for preventing the degradation caused by the presence of said insects and/or mites in said agricultural product. The object of the present invention is not to clean a structure (silos, warehouses, chambers...), with residual materials for preventing the contamination of any product that may be kept or stored in said structure.

**[0016]** The present invention is not restricted to an end product and therefore a raw product may also be treated with the claimed process. Then, the product may be stored until the moment of its distribution or until further processing is required.

**[0017]** In this invention, "eradication of insects and/or mites at any stage of development" means the elimination of insects at any of the following stages of development: egg, larva or nymph, pupa and adult.

**[0018]** In this invention, "container" refers to any package or place, like a silo, wherein the agricultural product is susceptible to be introduced. The container used in the process of this invention should have barrier properties to gases so that the agricultural product be exposed to the mixture of gases during step c), and the selection of the type can be made between the large variety of containers for packaging agricultural products.

**[0019]** As mentioned above, the process described in this invention permits the eradication of the insects and/or mites associated with the agricultural product, independently of the stage of development of said insects and/or mites, with the product reaching the final user in an optimum condition.

**[0020]** The main advantageous aspect of the process of this invention lies in the fact that the process substantially reduces the exposure time. Surprisingly, the oxygen acts as an enhancer of the eradicating effect of the mixture of gases. Furthermore, there is a critical range of oxygen gas in the mixture, where this surprising effect is highlighted. With this time reduction using the mixture of gases of the invention the manufacturers do not need to immobilize the product in the warehouse for long time, waiting for the effectiveness of the treatment. The product is ready to be sent to the supply chain in few hours or days and with the guarantee that no insects/mites exist inside the product.

## **FIGURES**

### **[0021]**

Figure 1 shows the time-to-death curves for 70% CO<sub>2</sub>, and 70% CO<sub>2</sub> and 35 ppm SO<sub>2</sub> applied to adults of *S. oryzae*;

Figure 2 shows SO<sub>2</sub> residual content with 70% CO<sub>2</sub> (5% O<sub>2</sub>) and 3% SO<sub>2</sub> at 25°C;

Figure 3 shows SO<sub>2</sub> residual content from wheat flour fumigated with: 70% CO<sub>2</sub> (5% O<sub>2</sub>) and different [SO<sub>2</sub>]; Measurements made at after the treatment.

## **DETAILED DESCRIPTION OF THE INVENTION**

**[0022]** Particularly, the process for eradication of insects and/or mites in an agricultural product according to the present invention uses a mixture of gases comprising a concentration of CO<sub>2</sub> ranging from 40% to 95%, preferably from 50% to 95%, more preferably from 70% to 95%; with a concentration of O<sub>2</sub> ranging from 0.5% to 15%, preferably from 5.0% to 12.5%, more preferably from 5.0% to 10.5%; and with a concentration of SO<sub>2</sub> higher than 20 ppm, preferably ranging from 30 ppm to 5%, more preferably from 150 ppm to 3%. The modified atmosphere (MA) with the composition of the mixture of gases can be balanced preferably with N<sub>2</sub>, although other inert gases may be used with the same purpose.

**[0023]** Advantageously, the agricultural product is exposed to the mixture of gases for a time lower than 20 days, preferably lower than 15 days, more preferable lower than 5 days, and still more preferably lower than 1 day in order to eradicate the insects and/or mites.

**[0024]** Surprisingly, the oxygen acts as an enhancer of the eradicating effect of the mixture of gases for eradicating insects and/or mites at any stage of their development in an agricultural product. Different trials were performed to show this effect. See Table 1.

**[0025]** A further surprising effect according to the process of the present invention is that the addition of a low concentration of SO<sub>2</sub> in a modified atmosphere (CO<sub>2</sub> and O<sub>2</sub>) increases the mortality of different types of pests in all stages of development when this process of eradication is compared to the mortality obtained without SO<sub>2</sub>. Therefore, the

presence of SO<sub>2</sub> in a MA allows reducing the exposure time for eradicating a pest.

**[0026]** Moreover, the process of the present invention establishes for the first time that the presence of SO<sub>2</sub> in a mixture of gases of CO<sub>2</sub> and O<sub>2</sub> seems to have a strong effect on the mortality of a pest which allows to significantly reducing the exposure time for eradicating a pest from an agricultural product, specially in certain ranges of CO<sub>2</sub> concentration and O<sub>2</sub> concentration (see, for example, Table 3).

**[0027]** The process described in the present invention is particularly useful for the control of a wide variety of insects or mites of agricultural products. Particular insects treated are *Sitophilus oryzae*, *Tribolium confusum* and *Plodia interpunctella*.

**[0028]** Now, the invention will be described in more detail making reference to the specific embodiments which are not intended to limit the scope of protection defined in the appended claims.

## **PREFERRED EMBODIMENTS OF THE INVENTION**

### **Operating conditions**

**[0029]** Insects used for the experiments came from colonies maintained on food diets at IRTA (Cabrils, Barcelona). Experiments were conducted with adults, larvae, pupae and eggs. All laboratory studies were conducted in a climatic chamber at 25±1°C, 70±10% RH and atmospheric pressure.

**[0030]** The experimental arenas were ventilated plastic cages (7.5 cm diameter and 3 cm high) containing 50g of food diet. The sides of the arenas were painted with *Fluon*<sup>TM</sup> in order to avoid the escape of insects during the treatment.

**[0031]** Fifty individuals were added to arenas. For obtaining eggs of *S. oryzae*, adult weevils (1280 adults/kg of brown rice) were added and allowed to oviposit for 7 days. Afterwards, adults were removed with a 2.0 mm mesh sieve and rice with eggs was used for the experiments.

**[0032]** Insects were exposed to each gas mixture in sealed glass desiccators (200 mm diameter). Three cages of each stage were exposed in the same desiccators and five replications were made for each gas mixture and exposure time according to each experiment. Gas mixtures consist of 70% CO<sub>2</sub> and 95% CO<sub>2</sub>, combined with SO<sub>2</sub> (0%, 35 ppm, 50 ppm, 150ppm and 3%) and 5% O<sub>2</sub>, balanced with N<sub>2</sub>.

**[0033]** Gas mixtures were previously prepared in cylinders and gas concentration verified by the company that made up the gases mixtures (Carburros Metálicos S.A.). Modified atmospheres were balanced with N<sub>2</sub>. Gas mixtures from the cylinders were continuously flushed into the desiccators at a pressure of 2 bars until the desired gas concentrations were reached. A gas analyzer (Abiss model TOM 12) was used to check the concentrations of CO<sub>2</sub> and O<sub>2</sub> inside the desiccators during the experiment.

**[0034]** After the treatment, desiccators were opened and cages were kept in a climatic chamber at 25±2°C of temperature, 70±10% R.H. and atmospheric pressure. To evaluate mortality on adults, the number of living adults in each cage was counted after 24-h. The percentage of mortality was calculated using the initial number of individuals placed in each cage. Sets of control cages were used to determine the percentage of natural mortality. For larvae, pupae and eggs, emerging progeny was counted after 4 to 9 weeks, depending on insect species and developmental stage. The number of living adults in each arena was counted and the percentage of mortality was calculated using the number of individuals emerged in the control cages. Results were subjected to one-way analysis of variance (ANOVA)(SAS System for Windows 8.02. SAS Institute, Cary, NC. 2001). Time-mortality data were subjected to Probit analysis by the POLO-PC computer program (LeOra Software Polo Plus 1.0 2002-2007) to determine LT50, and their respective 95% confident intervals.

### **SO<sub>2</sub> sorption and desorption in samples of rice, wheat flour and almonds**

**[0035]** The experimental arenas were glass Petri dishes (9 cm diameter and 2 cm high) containing 25 g of three different food products: polished rice, wheat flour, and almonds. Three different gas mixtures were used: 70% CO<sub>2</sub> with 50ppm SO<sub>2</sub>, 70% CO<sub>2</sub> with 150ppm SO<sub>2</sub>, and 70% CO<sub>2</sub> with 3% SO<sub>2</sub>. All gas mixtures contained 5% of O<sub>2</sub> and were balanced with N<sub>2</sub>.

**[0036]** Products were exposed to the modified atmosphere in sealed crystal desiccators (200 mm diameter). Three Petri dishes were exposed in the same desiccator to each gas mixture, except in the case of the mixtures containing 3% SO<sub>2</sub> where only one Petri dish was exposed in each desiccator. Four different exposure times were tested: 1, 2, 3 and 7 days. Three desiccators were used for each product and exposure time. Gas mixtures were previously prepared in cylinders and gas concentration verified as in the previous experiments. For the treatment, gas mixtures were continuously flushed through the desiccators for 2 minutes at a pressure of 2 bars. Desiccators were then kept in a climatic chamber at 25±1°C. After the treatment, a gas analyzer (Abiss model TOM 12) was used to check CO<sub>2</sub> and O<sub>2</sub> levels inside desiccators. SO<sub>2</sub> content in the food product was determined by the method of *Monier-Williams*. Analysis were conducted immediately after opening desiccators, and 3-h, 24-h and 7 days after the end of the treatment. Treated

samples were held in the laboratory at room temperature. SO<sub>2</sub> levels in untreated control samples were also analysed in three samples of rice and wheat flour, and six samples of almonds.

#### Insect mortality

**[0037]** When exposed to a MA of 70% CO<sub>2</sub>, the mortality rates of *S. oryzae*, *P. interpunctella* and *T. confusum* were strongly influenced by the addition of SO<sub>2</sub> (Table 2). Adding SO<sub>2</sub> significantly increased the percentage mortality up to from 83 to 100% when combined with 3% SO<sub>2</sub>. When increasing the CO<sub>2</sub> concentration from 70% to 95%, mortality in the treatments without SO<sub>2</sub> did not increased significantly. Again the addition of SO<sub>2</sub> resulted in a significant increase in mortality (100% when combined with 3% SO<sub>2</sub>) compared to the treatment without SO<sub>2</sub> (Table 2)

**[0038]** The mortality of adults exposed to a MA of 70% CO<sub>2</sub>, 5% O<sub>2</sub> with and without the 35ppm of SO<sub>2</sub> for a range of exposure times are given in Fig. 1. Time-to-death curves show that mortality of adults was higher for the range of 20-h to 48-h in presence of SO<sub>2</sub>, although variability observed was high, mainly after 20 hours exposure. At the LT<sub>50</sub> level, adults exposed to the combination of SO<sub>2</sub> and CO<sub>2</sub> were more susceptible by approximately 6-h, than adults exposed to CO<sub>2</sub> alone (Table 3).

**Table 1.** % Mortality of *S. oryzae* adults exposed to CO<sub>2</sub>, O<sub>2</sub>, and SO<sub>2</sub> during 24 hours and 25°C of temperature.

|  |                      |                     | % Mortality <i>S. oryzae</i> adults |
|--|----------------------|---------------------|-------------------------------------|
|  | % [CO <sub>2</sub> ] | % [N <sub>2</sub> ] | 150 ppm SO <sub>2</sub>             |
|  | 100                  | 0                   | 0                                   |
|  |                      |                     | 6.4±3.45                            |
|  | 95                   | 5                   | 0                                   |
|  |                      |                     | 8.0±1.20                            |
|  | 95                   | 0                   | 5                                   |
|  |                      |                     | 66.5±3.04                           |
|  | 90                   | 10                  | 0                                   |
|  |                      |                     | 6.6±2.30                            |
|  | 90                   | 0                   | 10                                  |
|  |                      |                     | 76.9±2.00                           |

**Table 2.** Critical composition of 70% and 95% of CO<sub>2</sub>, 50ppm to 3% of SO<sub>2</sub>, and 5% O<sub>2</sub> to eradicate three insect pest species.

|                    |                 | <i>S. oryzae</i> |            |            | <i>P. Inter.-punctella</i> | <i>T. confusum</i> |            |
|--------------------|-----------------|------------------|------------|------------|----------------------------|--------------------|------------|
|                    |                 | Adults           | Lar./pupae | Eggs       | Larvae                     | Adults             | Pupae      |
|                    | SO <sub>2</sub> | 24h              | 3d         | 3d         | 4h                         | 24h                | 6d         |
| 70%CO <sub>2</sub> | 0ppm            | 41,71±3,38       | 32,2±2,92  | 63,69±1,29 | 65,4±3,46                  | 15, 8±1,           | 98,81±0,38 |
|                    | 50ppm           | 49,18±5,08       | 29,8±1,45  | 92,42±0,86 | 67,7±3,13                  | 7,5±1,8            | 98,93±0,34 |
|                    | 150ppm          | 48,22±8,72       | 31,4±1,74  | 97,8±0,4   | 81,07±4,49                 | 12,05±0,9          | 99,75±0,25 |
|                    | 3%              | 100              | 82,7±1,83  | 100        | 100                        | 100                | 100        |
| 95%CO <sub>2</sub> | 0ppm            | 54.4±3.71        | 23,8±4,78  | 60,3±2,07  | 25,2±1,42                  | 45,8±4,32          | 100        |
|                    | 50ppm           | 52.0±3.13        | 22,3±2,16  | 63,9±2,    | 52,5±2,63                  | 37,1±2,18          |            |
|                    | 150ppm          | 66.5±3.04        | 16,66±2,97 | 69,2±1,94  | 48,4±2,05                  | 42,2±1,95          | 100        |
|                    | 3%              | 100              | 100        | 100        | 100                        | 100                | 100        |

**Table 3.** Mortality responses (LT<sub>50</sub>) of *S. oryzae* adults to 70% CO<sub>2</sub> (+5% O<sub>2</sub>) and 70% CO<sub>2</sub> and 35 ppm SO<sub>2</sub> (+5% O<sub>2</sub>) resulting from laboratory fumigations at 25°C

| Treatment                                                      | $\chi^2$ (d.f.) | Slope $\pm$ SE   | LT50 (hours) <sup>a</sup> |
|----------------------------------------------------------------|-----------------|------------------|---------------------------|
| 70% CO <sub>2</sub>                                            | 805.93 (88)     | 10.71 $\pm$ 0.33 | 26.8 (25.7-28.0)          |
| 70% CO <sub>2</sub> + 35ppm SO <sub>2</sub>                    | 3202.3 (103)    | 8.99 $\pm$ 0.36  | 20.4 (17.5-22.4)          |
| <sup>a</sup> numbers in brackets give the 95% confidence range |                 |                  |                           |

#### Analysis of sulphur dioxide residues, specially for food products

**[0039]** The initial residues of SO<sub>2</sub> detected in almonds, wheat flour and rice before the treatments were 3.6  $\pm$  1.85ppm, 23.6  $\pm$  12.80ppm, and 0ppm, respectively.

**[0040]** When treated with a MA of 3% SO<sub>2</sub>, different sorption curves were observed for the three food products during the assay. Wheat flour reached the highest values (aprox. 3500ppm), and remained more or less stable from the third day of analysis onwards (Fig. 2). SO<sub>2</sub> adsorption of both almonds and rice steadily increased from the first day until the end of the study (to approx. 2300ppm). After aeration, a high degree of desorption was observed for wheat flour treated with 3% SO<sub>2</sub> (Fig 3). Values dropped from more than 2500ppm just after the treatment to around 400ppm after 7 days ventilation.

**[0041]** When treated with a low SO<sub>2</sub> content (150ppm and 50ppm), initial residual contents of SO<sub>2</sub> after the treatment were low. After 3-h and 24-h ventilation, the residual limits also decreased quickly even to the minimum detection limit of the technique.

**[0042]** In conclusion, the addition of a low concentration of SO<sub>2</sub> (from 35 ppm to 3%) in a MA of 70% and 95% CO<sub>2</sub> increased the mortality of *S. oryzae*, *P. interpunctella* and *T. confusum* compared to the mortality obtained with the same MA without SO<sub>2</sub>. The highest level of SO<sub>2</sub> tested (3%) had a strong effect on mortality, while concentrations of 150ppm SO<sub>2</sub> or lower had less impact on mortality. Residual concentrations of SO<sub>2</sub> in wheat flour were low after the 150ppm and 50ppm SO<sub>2</sub> treatments. After 7 days aeration residual levels in all treatments were low or undetectable.

#### Claims

1. Process for eradication of insects or mites at any stage of their development in an agricultural product, **characterized in that** it comprises the steps of:

- a) placing the agricultural product inside of a container;
- b) filling the container with a mixture of gases consisting of CO<sub>2</sub>, O<sub>2</sub>, SO<sub>2</sub>, and an inert gas, wherein the concentration of the CO<sub>2</sub> is higher than 40%, the O<sub>2</sub> is between 0.5 to 10.5%, the SO<sub>2</sub> is between 50 ppm to 3%, and balanced with an inert gas, by means of flushing the mixture of gases or by means of creating a vacuum inside the container then filling the container with said mixture;
- c) exposing the agricultural product to the mixture of gases for a time that ranges between 4 hours and 20 days, so that it permits the eradication of insects and/or mites at any stage of their development inside of the container.

2. Process for eradication according to claim 1, wherein CO<sub>2</sub> is between 70 to 95%, O<sub>2</sub> is between 5 to 10.5%, and SO<sub>2</sub> is between 150ppm to 3%, and balanced with an inert gas.

3. Process for eradication according to claim 2, wherein said inert gas is nitrogen (N<sub>2</sub>).

4. Process for eradication according to claim 1, **characterized in that** steps b) and c) are carried out at atmospheric pressure and at room temperature.

5. Process for eradication according to claim 1, **characterized in that** said insect is *Sitophilus oryzae*.

6. Process for eradication according to claim 1, **characterized in that** said insect is *Tribolium confusum*.

7. Process for eradication according to claim 1, **characterized in that** said insect is *Plodia interpunctella*.

## EP 2 177 117 A1

8. Process for eradication according to claim 1, **characterized in that** said agricultural product is a manufactured product.
9. Process for eradication according to claim 1, **characterized in that** said agricultural product is a cultivated product.
10. Process for eradication according to claim 1, **characterized in that** said agricultural product is a non-perishable food product.

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FIG. 1

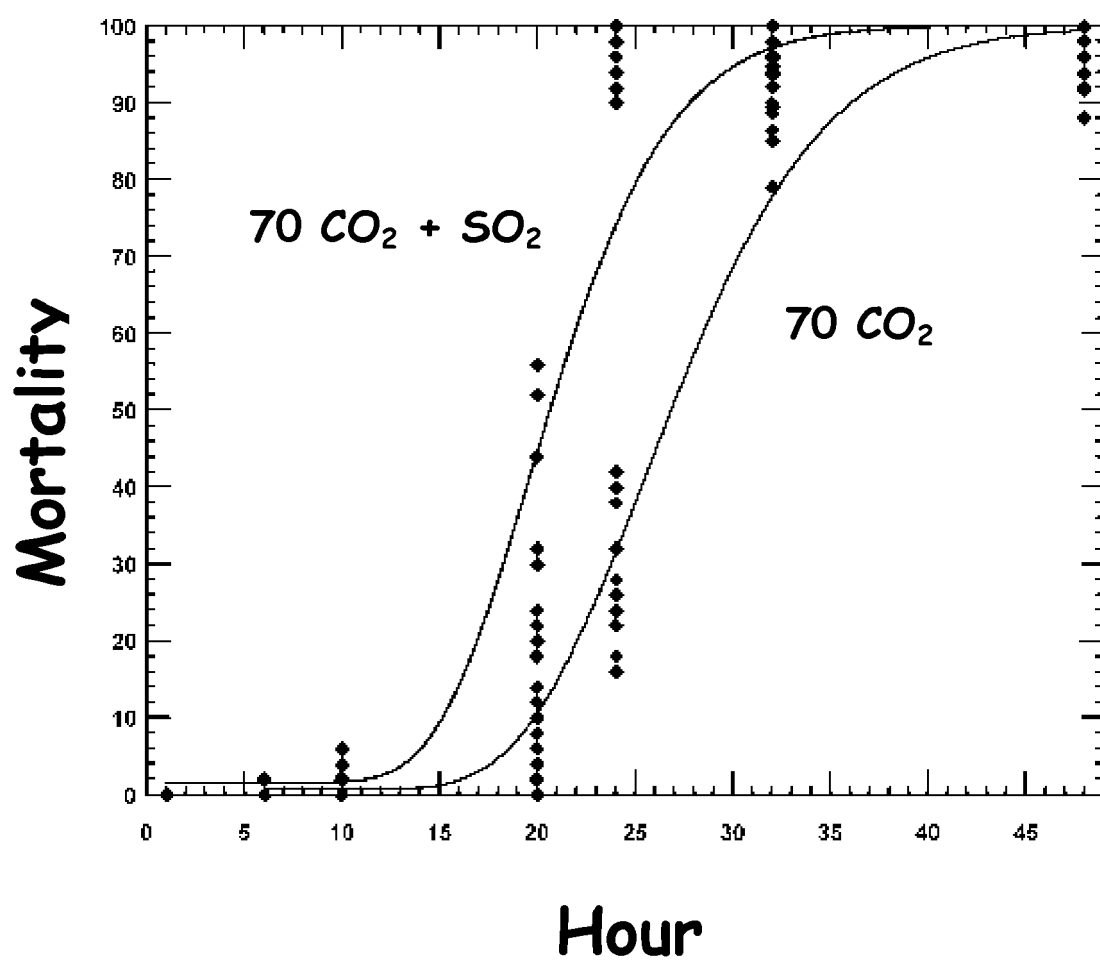
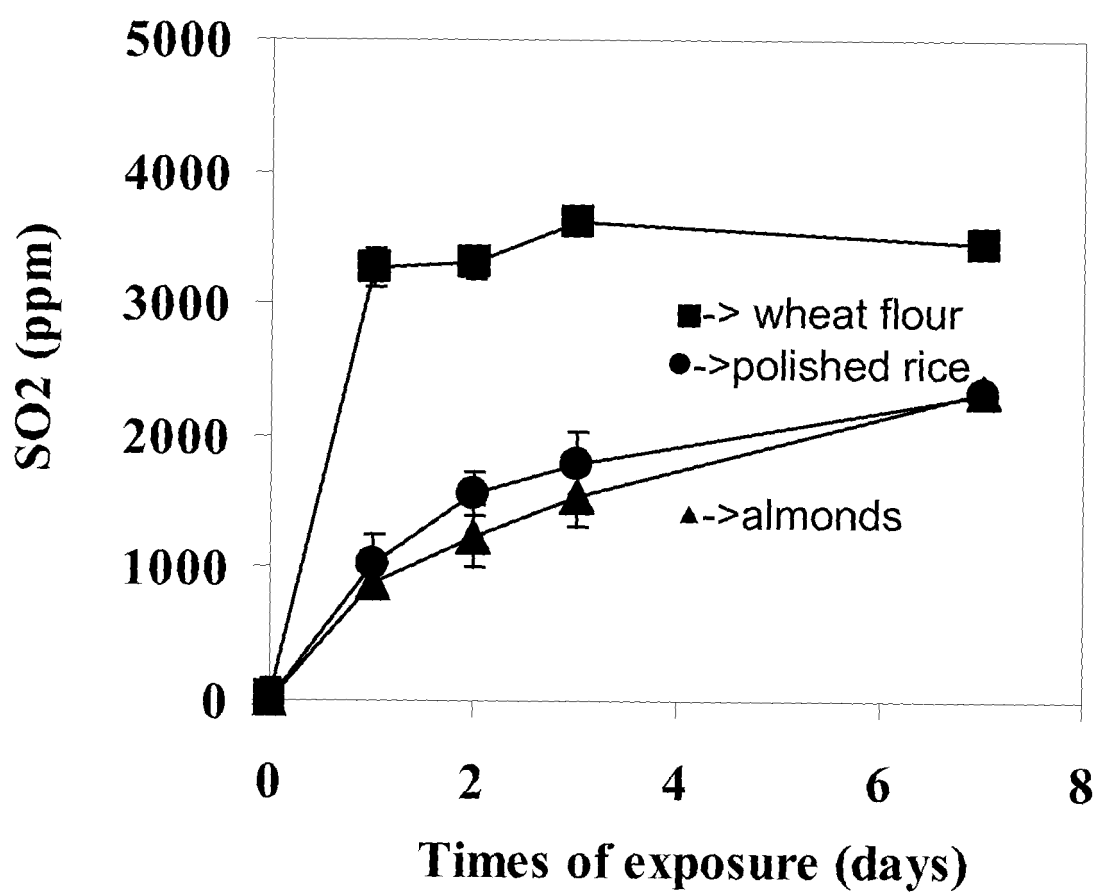
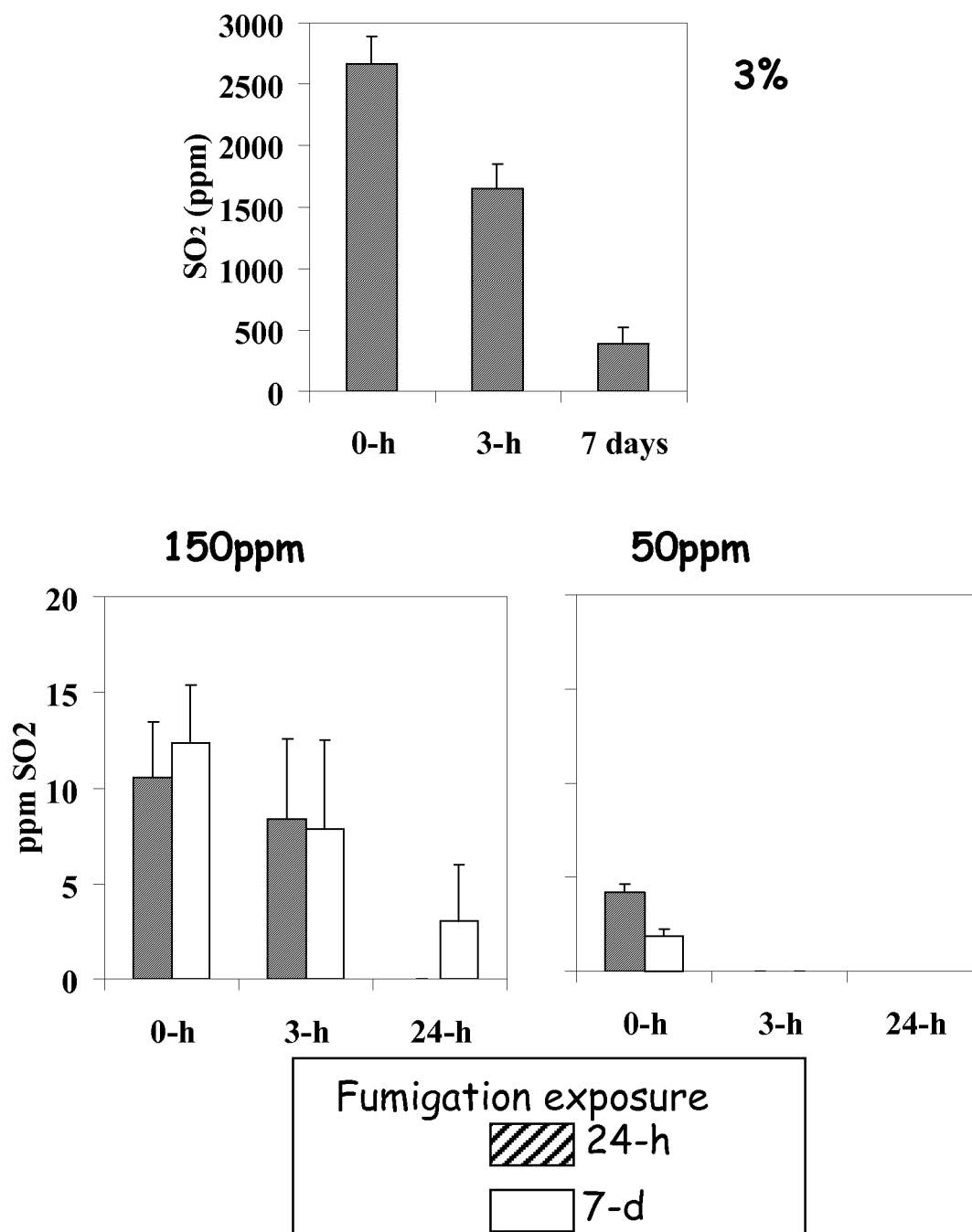


FIG. 2



**FIG. 3**





## EUROPEAN SEARCH REPORT

Application Number  
EP 09 17 0684

| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                            |                                                                                  |
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| Category                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | Citation of document with indication, where appropriate, of relevant passages                                                                                                                                                                                                                                                                                                                                                                                                                                     | Relevant to claim                                          | CLASSIFICATION OF THE APPLICATION (IPC)                                          |
| Y                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | <p>DATABASE BIOSIS [Online]<br/>BIOSCIENCES INFORMATION SERVICE,<br/>PHILADELPHIA, PA, US;<br/>1991,<br/>FLEURAT-LESARD F ET AL: "THE INFLUENCE OF<br/>OXYGEN CONTENT AT ATMOSPHERES ENRICHED<br/>WITH CARBON DIOXIDE ON THE MORTALITY OF<br/>HIDDEN STAGES OF SITOPHILUS-SPP IN WHEAT<br/>GRAINS"<br/>XP002565924<br/>Database accession no. PREV199191105886<br/>* abstract *</p> <p>&amp; ENTOMOLOGIA EXPERIMENTALIS ET APPLICATA,<br/>vol. 58, no. 1, 1991, pages 37-48,<br/>ISSN: 0013-8703</p> <p>-----</p> | 1-10                                                       | <p>INV.<br/>A23L3/3445<br/>A23B9/22<br/>A01N59/02<br/>A01N59/04<br/>A01P7/04</p> |
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| The present search report has been drawn up for all claims                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                            |                                                                                  |
| Place of search<br><b>Munich</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | Date of completion of the search<br><b>29 January 2010</b> | Examiner<br><b>Molina de Alba, José</b>                                          |
| <p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone<br/>Y : particularly relevant if combined with another document of the same category<br/>A : technological background<br/>O : non-written disclosure<br/>P : intermediate document</p> <p>T : theory or principle underlying the invention<br/>E : earlier patent document, but published on, or after the filing date<br/>D : document cited in the application<br/>L : document cited for other reasons</p> <p>&amp; : member of the same patent family, corresponding document</p> |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                            |                                                                                  |

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EPO FORM 1503 03.82 (P04C01)



## EUROPEAN SEARCH REPORT

Application Number  
EP 09 17 0684

| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                     |                                         |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------|-----------------------------------------|
| Category                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | Citation of document with indication, where appropriate, of relevant passages                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Relevant to claim                                   | CLASSIFICATION OF THE APPLICATION (IPC) |
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| The present search report has been drawn up for all claims                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                     |                                         |
| Place of search<br>Munich                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | Date of completion of the search<br>29 January 2010 | Examiner<br>Molina de Alba, José        |
| CATEGORY OF CITED DOCUMENTS<br>X : particularly relevant if taken alone<br>Y : particularly relevant if combined with another document of the same category<br>A : technological background<br>O : non-written disclosure<br>P : intermediate document<br>T : theory or principle underlying the invention<br>E : earlier patent document, but published on, or after the filing date<br>D : document cited in the application<br>L : document cited for other reasons<br>& : member of the same patent family, corresponding document |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                     |                                         |

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EPO FORM 1503 03.02 (P04C01)



## EUROPEAN SEARCH REPORT

Application Number  
EP 09 17 0684

| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                                                                                                                                                                                                                                                                                                                                       |                                                     |                                         |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------|-----------------------------------------|
| Category                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | Citation of document with indication, where appropriate, of relevant passages                                                                                                                                                                                                                                                                                         | Relevant to claim                                   | CLASSIFICATION OF THE APPLICATION (IPC) |
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| The present search report has been drawn up for all claims                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                                                                                                                                                                                                       |                                                     |                                         |
| Place of search<br>Munich                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                                                                                                                                                                                                                                                                                                                                       | Date of completion of the search<br>29 January 2010 | Examiner<br>Molina de Alba, José        |
| <p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone<br/>Y : particularly relevant if combined with another document of the same category<br/>A : technological background<br/>O : non-written disclosure<br/>P : intermediate document</p> <p>T : theory or principle underlying the invention<br/>E : earlier patent document, but published on, or after the filing date<br/>D : document cited in the application<br/>L : document cited for other reasons<br/>.....<br/>&amp; : member of the same patent family, corresponding document</p> |                                                                                                                                                                                                                                                                                                                                                                       |                                                     |                                         |

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EPC FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 09 17 0684

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

29-01-2010

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**REFERENCES CITED IN THE DESCRIPTION**

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